भारतीय मानक
Indian Standard

IS 3859 : 2019

वस्त्र रंजक सामग्री — टाईटेनियम ट्राईक्लोराइड के संग अपचयन द्वारा जल-विलेय एज़ो रंजको की तीव्रता के निर्धारण की विधि

(पहला पुनरीक्षण)

Textile Dyestuffs — Method for Determination of Strength of Water-Soluble Azo Dyes by Reduction with Titanium Trichloride

(First Revision)

ICS 59.040

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Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Textile Speciality Chemicals and Dyestuffs Sectional Committee had been approved by the Textiles Division Council.

This standard was first published in 1966 and the present revision has been taken up to update the general information about analysis of azo dyes given in Annex A. Water soluble azo (direct and acid) dyes are available in the market generally in diluted form. These dyes are usually checked by dyeing method against standard samples as agreed to between the buyer and the seller. These standard samples are usually designated arbitrarily as hundred percent. This creates difficulties in reporting records of production, import-export statistics or comparing dyes where due to slight shade differences the assessment of strength by dyeing method becomes difficult.

This method is mainly useful for production control, production statistics and import-export statistics, where one normally deals with unblended dyestuffs which are straight dilutions of crude dyes and where one requires an agreed method of test for comparison.

The method outlined in this standard is useful for comparing the strengths of dyestuffs which have slight shade differences but have the same colour index number. In case of mixtures, this method may be useful provided the blending composition of the mixtures is the same. However, the strength calculated will be an arbitrary figure because of the differing molecular weights of dyes used in the mixtures.

The composition of the Committee responsible for the formulation of this standard is given at Annex C.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

TEXTILE DYESTUFFS — METHOD FOR DETERMINATION OF STRENGTH OF WATER-SOLUBLE AZO DYES BY REDUCTION WITH TITANIUM TRICHLORIDE

(First Revision)

1 SCOPE

This standard prescribes a method for determination of strength of water-soluble azo dyes listed in Annex A. The method prescribed in the standard is not applicable to mixtures of dyestuffs and the dyestuffs containing reducible impurities.

2 REFERENCES

The following standard contains provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below.

IS No.	*Title*
1070 : 1992	Reagent grade water

3 PRINCIPLE

The azo dye is reduced by titanium trichloride. Knowing the amount of titanium trichloride used for reducing the dye, the strength of dye is calculated.

4 SAMPLING

4.1 Lot

All the containers of the same dye and the same concentration delivered to a buyer against a despatch note shall constitute a lot.

4.2 Unless otherwise agreed to between the buyer and the seller the number of containers to be selected at random from a lot shall be in accordance with Table 1.

4.3 From each container draw small quantities of the dye by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

Table 1 Sample Size

(*Clause* 4.2)

Lot Size (1)	Sample Size (2)
Up to 100	3
101- 300	4
301- 500	5
501 and above	7

5 APPARATUS

5.1 Titanium Trichloride ($TiCl_3$) Storage Bottle — *see* Fig. 1.

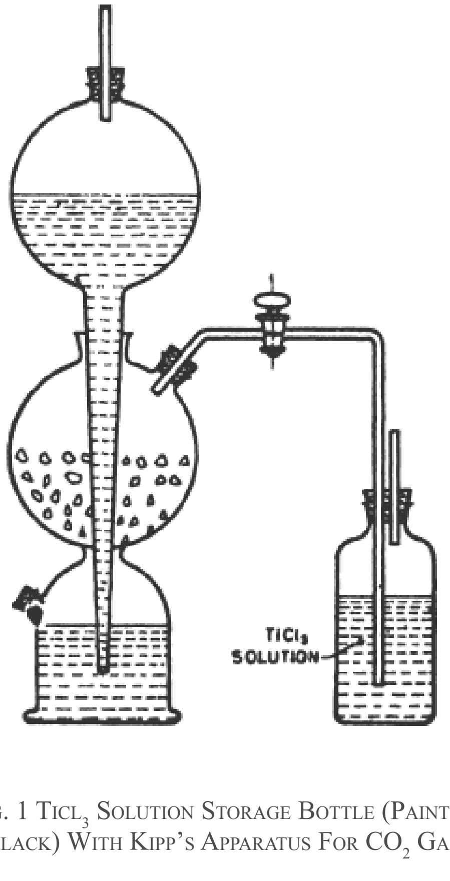

FIG. 1 $TiCl_3$ SOLUTION STORAGE BOTTLE (PAINTED BLACK) WITH KIPP'S APPARATUS FOR CO_2 GAS

5.2 Reduction Flask — *see* Fig. 2.

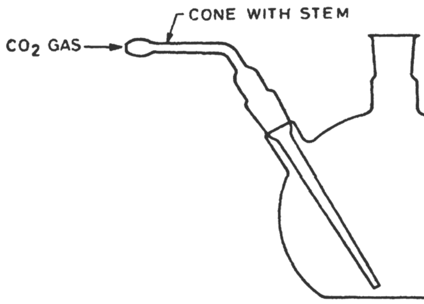

FIG. 2 500 ML REDUCTION FLASK (TWO NECKS, SHORT CENTRE NECK AND SIDE NECK AT AN ANGLE)

6 REAGENTS

6.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS 1070 : 1992) shall be used where the use of water or distilled water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

6.1 Hydrochloric Acid

a) Concentrated, and
b) 25 percent (w/v).

6.2 Ammonium Thiocyanate Solution, 20 percent (w/v).

6.3 Titanium Trichloride Solution

Approximately 0.025 N, prepared as follows:

Prepare a 15 percent (w/v) solution of titanium trichloride. Take 30 ml of the solution. Filter it through a thick pad of glass wool. Add this solution to a previously boiled mixture of one litre of water and 60 ml of concentrated hydrochloric acid. Mix this solution by passing into it a current of inert gas, such as carbon dioxide or nitrogen, for about 15 min. Store the mixture in a bottle in an atmosphere of carbon dioxide.

NOTE — Carbon dioxide may be supplied by Kipp's apparatus. The solution should be stored in a bottle painted black to protect it from light.

6.4 Stannous Chloride Solution

Prepared by dissolving 30 g of stannous chloride in 100 ml of concentrated hydrochloric acid and diluting it to 200 ml with water. Keep the solution in air-tight bottle.

NOTE — This solution should be prepared fresh after one week.

6.5 Mercuric Chloride Solution

Prepared by dissolving 5 g of mercuric chloride in 100 ml of water and filtered, if necessary.

6.6 Diphenylamine Solution

Prepared by melting 1 g of diphenylamine and adding to this 100 ml of concentrated sulphuric acid. Dissolve diphenylamine by stirring.

6.7 Sulphuric Acid, 1 N.

6.8 Orthophosphoric Acid, free from iron.

6.9 Potassium Dichromate Solution, 0.1 N.

6.10 Ferric Ammonium Sulphate Solution

Approximately 0.025 N, prepared as follows:

Dissolve 9.803 g of pure ferrous ammonium sulphate [$FeSO_4.(NH_4)2SO_4.6\ H_2O$] in 100 ml of water. Add 10 ml of concentrated sulphuric acid and mix well. Dissolve 0.79 g of potassium permanganate in 100 ml of water. Add this solution to the solution of ferrous ammonium sulphate with stirring. At the end, add the permanganate solution carefully, so that a ferrous salt is just oxidized. Cool the solution and dilute it to one litre. Determine the normality of the solution by the method given in Annex B.

7 PROCEDURE

7.1 Weigh accurately 1 g of dyestuff from the test sample. Dissolve it in hot water. Pour the solution into a 500 ml graduated flask and make the volume to 500 ml. Mix well.

NOTE — In the case of some azo dyes (*see* Annex A) 0.5 g of dyestuff should be taken for the test.

7.2 Pipette 50 ml of the solution into a 500 ml reduction flask containing 50 ml of dilute hydrochloric acid and 50 ml of water which have been previously brought to boil. Start passing carbon dioxide into the solution and simultaneously heat the contents to boil.

7.3 Pass carbon dioxide in the storage bottle containing titanium trichloride solution and immediately draw 50 ml of titanium trichloride solution by a pipette in which carbon dioxide has been passed previously. Add this solution to the dye solution (*see* **7.2**). Immediately boil the contents of the flask for complete reduction (*see* Note 1). Cool the flask in ice-bath. Maintain the continuous flow of carbon dioxide throughout the test. Add 10 ml of 20 percent ammonium thiocyanate indicator solution. Titrate the mixture against 0.025 N ferric ammonium sulphate solution to a permanent change of colour. Note the reading of ferric ammonium sulphate solution (*see* Note 2).

NOTES:

1 The reduction time for various dyes is given in Annex A.

2 The volume of ferric ammonium sulphate required for titration, should not be less than 10 ml.

7.4 Carry out a blank by following the procedure given as above using 50 ml of water in place of dye solution.

7.5 Calculate the titanium value, in percent, of the dye by the following formula:

$$T = \frac{(B - A) \times N \times M \times V}{E \times C \times W \times 10}$$

Where,

T = Titanium value, percent, of the dye;

B = Volume in ml, of ferric ammonium sulphate solution required for blank (*see* **7.4**);

A = Volume in ml, of ferric ammonium sulphate solution required for the dye (*see* **7.3**);

N = Normality of ferric ammonium sulphate solution (*see* **B-1.3**);

M = Molecular weight of dye;

V = Volume of dye solution (*see* **7.1**);

E = Number of hydrogen atoms consumed per molecule of dye (*see* Note);

C = Volume in ml, of aliquot taken for titration (*see* **7.2**); and

W = Weight in g, of the sample taken for test.

NOTE — Each azo group requires 4, nitro group 6 and hydrozo group 2 atoms of hydrogen for reduction.

7.6 Repeat the test prescribed in **7.3** twice and calculate the titanium value, in percent, of the dye.

7.7 Calculate the average of the values obtained as in **7.5** and **7.6**.

8 REPORT

Report the titanium value obtained as in **7.7** as the strength of the dye under test.

ANNEX A

(*Clause* 1, *Notes under* 7.1 *and* 7.3)

GENERAL INFORMATION ABOUT ANALYSIS OF AZO DYES

Sl No. (1)	Colour Index* Number (2)	Name of Dye (3)	Molecular Weight of Dye as Acid (4)	Hydrogen Equivalent (5)	Concentration of Dye in g/500 ml (6)	Reduction Time in Minutes (7)
i)	13065	Acid yellow 36	353	4	1	12
ii)	14720	Acid red 14	458	4	1	10
iii)	15510	Acid orange 7	328.5	4	1	10
iv)	15620	Acid red 88	378	4	1	12
v)	16230	Acid orange 10	408	4	0.5	10
vi)	16255	Acid red 18	538	4	1	10
vii)	18055	Acid violet 7	522	4	1	10
viii)	20470	Acid black 1	572	14	0.5	10
ix)	24895	Direct yellow 12	636	8	1	8
x)	26370	Acid black 24	701	8	0.5	13
xi)	27855	Direct violet 7	659	8	0.5	12
xii)	27905	Direct violet 51	675	8	1	8
xiii)	29100	Direct red 31	669	8	0.5	8
xiv)	29150	Direct orange 26	712	8	1	15
xv)	29160	Direct red 23	769	8	1	8

* Colour Index. 1956. Edition 2. Society of Dyers and Colourists and the American Association of Textile Chemists and Colorists.

ANNEX B

(*Clause* 6.10)

DETERMINATION OF NORMALITY OF FERRIC AMMONIUM SULPHATE

B-1 PROCEDURE

B-1.1 Pipette 100 ml of ferric ammonium sulphate solution (*see* **6.10**) into a 500 ml conical flask and add 5 ml of concentrated hydrochloric acid. Heat it to about 85°C. Add stannous chloride solution dropwise from a burette while swirling the flask over a white background until the yellow colour of the solution has nearly disappeared. Complete the reduction by adding dilute solution of stannous chloride (*see* Note 1) dropwise with agitation after each addition until the liquid has a faint green colour free from any tinge of yellow. Dilute the solution in the flask to thrice its volume with water. Rapidly cool the solution under the tap with stopper on and remove the excess of stannous chloride by adding 10 ml of mercuric chloride solution rapidly in one lot. A slight silky white precipitate of mercurous chloride is obtained (*see* Note 2). Add 50 ml of 1 N sulphuric acid 2.5 ml of orthophosphoric acid and three drops of diphenylamine solution as indicator. Titrate slowly against 0.1 N potassium dichromate solution while stirring constantly to the first permanent tinge of purple or violet blue colouration.

NOTES:

1 Dilute stannous chloride solution should be prepared by diluting stannous chloride solution (*see* **6.4**) with two volumes of 10 percent hydrochloric acid.

2 Excess of stannous chloride solution should not be added. A heavy, grey or black precipitate is the indication of too much stannous chloride solution. Discard the solution and start with a fresh solution of ferric ammonium sulphate.

B-1.2 Carry out a blank by following the procedure prescribed in **B-1.1** but by using 100 ml of water in place of 100 ml of ferric ammonium sulphate solution.

B-1.3 Calculate the normality of ferric ammonium sulphate solution by the formula given below:

$$N = \frac{(A - B) \times N_1}{100}$$

Where,

N = Normality of ferric ammonium sulphate solution;

A = Volume in ml, of potassium dichromate solution required to titrate the solution (*see* **B-1.1**);

B = Volume in ml, of potassium dichromate solution required to titrate the blank (*see* **B-1.2**); and

N_1 = Normality of potassium dichromate solution.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

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Department for Jute and Fibre Technology Institute of Jute Technology University of Calcutta	Prof A. K. Samanta (***Chairman***)
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Atul Ltd (Colors Division), Atul	Shri Sai Ganesh Shri Arindam Chakraborty (*Alternate*)
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Wool Research Association, Thane	Smt Smita Bait Shri Chetan Mahajan (*Alternate*)
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